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Effect of nitrogen and nickel on the stability of an Fe–12Cr–23Mn austenitic steel

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The effect of nitrogen and nickel on the austenite stability and the structural changes as a result of cold work of an Fe–12Cr–23Mn alloy steel has been examined by transmission electron microscopy and X-ray analysis. Prestrain was performed either at room temperature or at 200° C. Final strain was carried out at liquid nitrogen temperature. The volume fraction of strain-induced martensite decreased by 40% and 12% due to the addition of 2.43% Ni and 0.11% N, respectively, despite whether prestrain was performed or not. The mechanisms are discussed in terms of the ε phase, stacking faults, dislocation cells and dislocation tangles induced by prestrain and final strain.

1. Introduction

Recently many works have been carried out to explore the possibility of the substitution of manganese and aluminium for nickel and chromium in austenite stainless steels. Some mechanical properties of various Fe–Al–Mn alloy steels were investigated by Schmatz [1] and Wan *et al.* [2]. Wang and Beck [3] discussed the Fe–30Mn–10Al–Si composition for sea-going ship propellers as a new stainless steel without nickel and chromium which has good resistance to marine corrosion. Fe–Mn–Cr systems of partial substitution were also examined [4, 5].

An extensive study on an Fe–12Cr–23Mn austenitic steel has been performed recently by the authors [5]. This study included ε(hcp) and α(bcc) martensitic transformation, prestrain effect on austenite stability, hardness and work-hardening behaviour. The strain-induced martensitic transformation has been an interesting research subject due to the close association of ε(hcp) and α(bcc) martensites [5–7]. The addition of nickel and nitrogen to Fe–Cr–Mn alloys will increase austenite stability because nickel and nitrogen are austenite formers and their addition can lower the M_s and M_d temperatures. In this work we investigated the effects of martensitic transformation and austenite stability in an Fe–12Cr–23Mn system due to the addition of nickel and nitrogen, respectively. It is hoped that through this study a better understanding of Fe–12Cr–23Mn systems can be obtained. This investigation may, in addition, pave the way for the improvement of austenite stability of Fe–Cr–Mn alloy steels.

2. Materials and methods

The chemical composition of the alloy steel investigated in this study is listed in Table I. Three types of alloys were examined. They were alloy NO (no nickel and no nitrogen), alloy 1N (containing about 0.1% nitrogen, but no nickel), and alloy NI (containing about 2.5% nickel but no nitrogen). The alloys were prepared by induction melting appropriate proportions of master alloys and alloying elements in an argon atmosphere. The ingots were forged, homogenized and cold-rolled to a thickness of 3 mm as described previously [5]. The homogenized plates with a final thickness of 3 mm were annealed at 950° C for 2 h followed by water quenching, which constituted the as-annealed condition.

Three types of thermomechanical processes were performed.

1. As-annealed → rolled at elevated temperature (200° C) to 20% or 40% thickness reduction (prestain) → rolled at − 196° C to 5, 10, 15 or 30% thickness reduction (final strain). This process was represented by E2-10, E4-30, etc. E2 before the dash stands for prestrained 20% at elevated temperature.

TABLE I Grain size and composition of alloy steels used (wt %)

Type of alloy	Av. grain diam. (μm)	Cr	Si	Al	Mn	Ni	N	C	S	P
1N	3.0	11.58	1.5	0.6	23	–	0.11	< 0.03	< 0.03	< 0.03
NI	3.9	12.01	1.55	0.7	23.6	2.43	–	< 0.03	< 0.03	< 0.03
NO	4.5	11.46	1.48	0.6	22.1	–	–	< 0.03	< 0.03	< 0.03

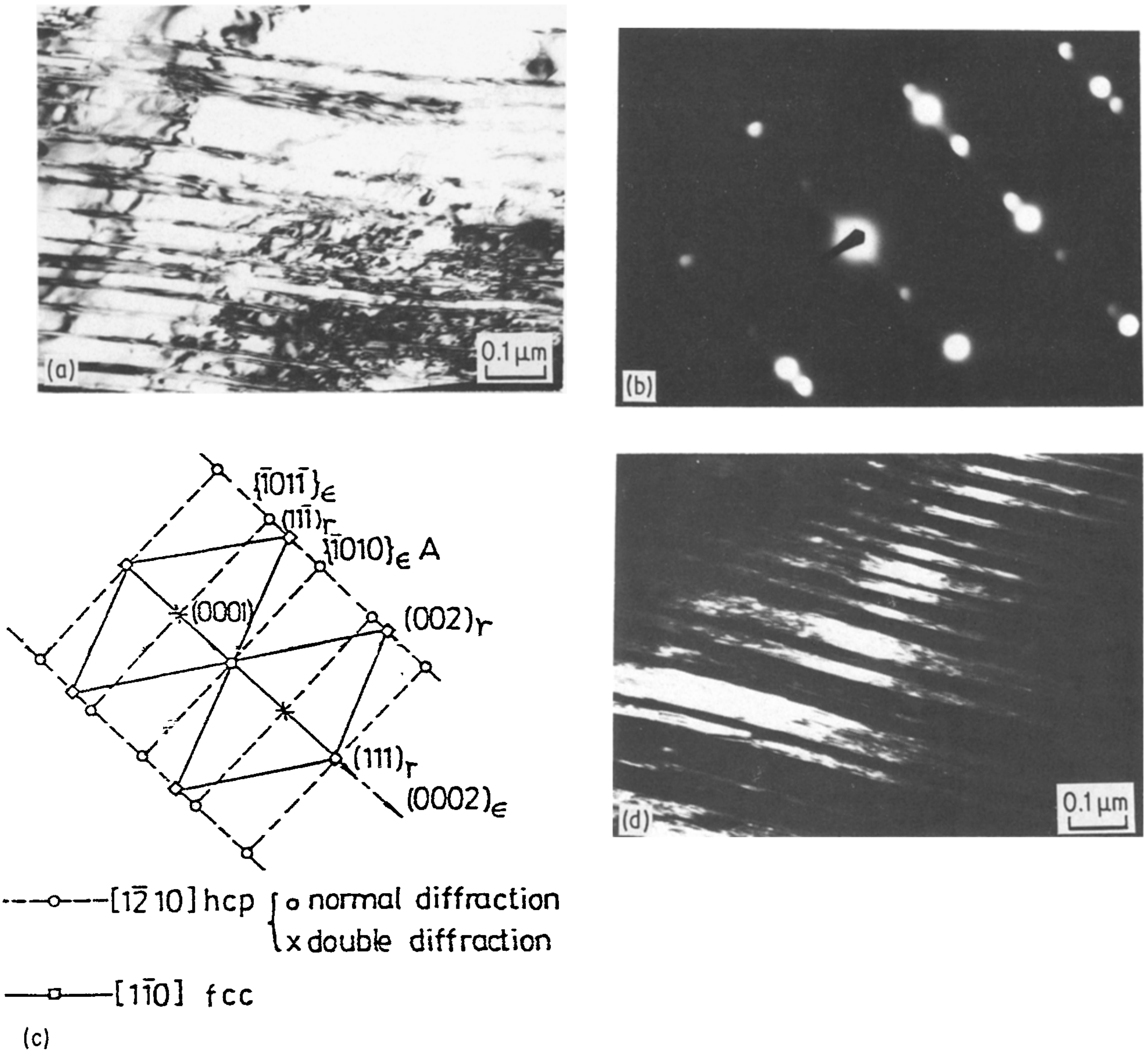

Figure 1 TEM microstructure of as-quenched specimen NO. (a) Bright-field image, (b) diffraction pattern taken at centre of (a), (c) idexed diffraction pattern, (d) dark-field image due to the reflection of $\{\bar{1}\,0\,1\,0\}_\varepsilon$.

Ten after the dash tells us the percentage of final strain at liquid nitrogen temperature.

2. As-annealed → rolled at room-temperature to 20% or 40% thickness reduction (prestrain) → rolled at −196° C to 5, 10, 15 or 30% thickness reduction (final strain). This process was indexed by R4-10, R2-15, etc. R4 before the dash means prestrained 40% at room temperature. The number after the dash gives the percentage of final strain at −196° C.

3. As-annealed → rolled at −196° C to 5, 10, 15 or 30% reduction in thickness. The process was represented by NP-30, NP-05, etc. NP means no prestrain. The number after the dash again tells the percentage of final strain performed at liquid nitrogen temperature. The specimens were indexed as NO-E2-05, 1N-R4-15, NI-NP-30, etc. The letters before the first dash indicated the type of alloy, the letters and number after the first dash represented the type of thermomechanical process.

Examination of microstructure was performed by optical and transmission electron microscopes (TEM). The martensitic transformation and austenite stability was examined by TEM and X-ray techniques. The details were described previously [5].

3. Experimental results

3.1. Martensitic transformation due to liquid nitrogen quenching

The phase of specimen at as-annealed condition was a full austenite regardless of alloy type. The grain size resolved by TEM was 4.5, 3.9 and 3.0 µm for alloy NO, NI and 1N, respectively. Some specimens were quenched from the annealing temperature (950° C) directly to liquid nitrogen which constituted the as-quenched condition. Neither ε(hcp) nor α(bcc) martensite was detected in as-quenched specimens of alloys 1N and NI through TEM and X-ray analysis. However, some very fine ε phases were detected in an as-quenched specimen of alloy NO as shown in Fig. 1. The phase was identified by selected-area diffraction pattern (Figs 1b and c). Fig. 1d shows the dark-field image due to the ε reflection of spot A of Fig. 1c. No α martensite was found in the as-quenched specimen of alloy NO. It is clear that M_s (temperature at which the martensite transformation starts spontaneously on cooling) of the ε phase of NI and 1N alloys is below −196° C. But M_s of the ε phase of alloy NO is above −196° C. M_s of α phase of alloys NI, 1N and NO is below −196° C.

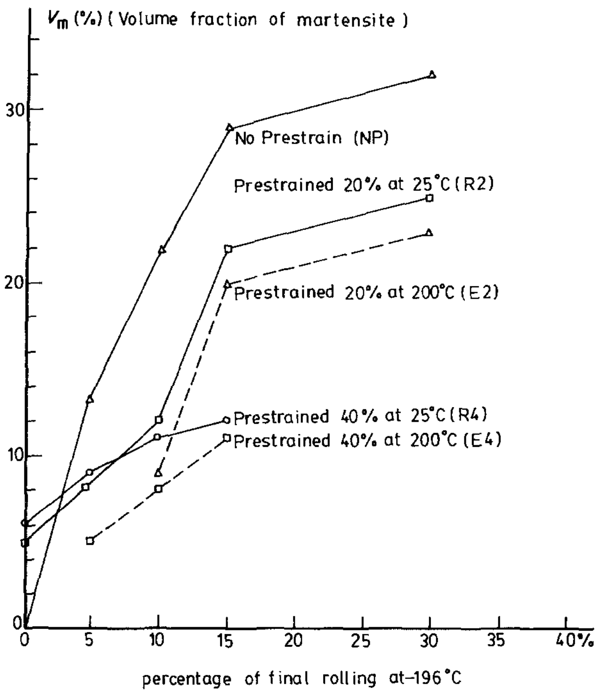

Figure 2 Correlation between the volume fraction of martensite and the amount of prestrain and final strain in specimen NO.

3.2. Strain-induced martensitic transformation

Both X-ray analysis and TEM investigation showed evidence of ε transformation in specimens deformed at 25° C or − 196° C regardless of alloy type. X-ray and TEM diffraction pattern indicated the presence of $10\bar{1}1$, $10\bar{1}2$ and $11\bar{2}0$ reflections which where typical of ε (h c p) structure [5]. Most of the ε phases were formed by overlapping of stacking faults. α martensite was found only after rolling the specimen at liquid nitrogen temperature regardless of alloy type. In no case was α-phase observed without the accompanying ε-phase. The identification of α-phase was described previously [5]. α-phases were found predominantly at the intersections of ε bands and stacking fault, grain boundary or another band. The amount of α-phase was always much less than ε-phase.

X-ray analysis was used to determine the volume fraction of α and ε martensite [5]. The relationship between the volume fraction of martensite and the amount of final rolling at − 196° C is shown by the curve NP in Figs 2, 3 and 4 for alloys NO, 1N and NI, respectively. From Figs 2 to 4, it is clear that alloy NO possesses the least austenite stability (highest amount of martensite) and alloy NI possesses the highest austenite stability (least amount of martensite transformation) with respect to straining. For example, the martensite volume fraction was 32%, 28% and 19%, respectively, in alloys NO, 1N and NI after rolling 30% at liquid nitrogen temperature.

3.3. Prestrain effect on austenite stability

X-ray analysis was used to determine the total volume fraction of ε and α martensite. The analysis was carried out by calculation of the ratio of the integrated intensity of a peak due to martensite (ε + α) to a peak due to the combination of austenite and martensite on the diffraction patterns [5]. The effect of prestrain and final strain on the martensite (α + ε) transformation is demonstrated by curves R2, E2, R4 and E4 in Figs 2, 3 and 4 for alloys NO, 1N and NI, respectively. It is clear that the general effects of prestrain and final strain on martensite transformation are similar for alloys NO, 1N and NI. They are: martensite volume fraction decreased as the prestrain was increased at the same amount of final strain; prestrain at 200° C showed more pronounced effect on inhibiting martensite transformation than prestrain at room temperature; and the rate of martensite transformation decreased either as the prestrain or final strain was increased.

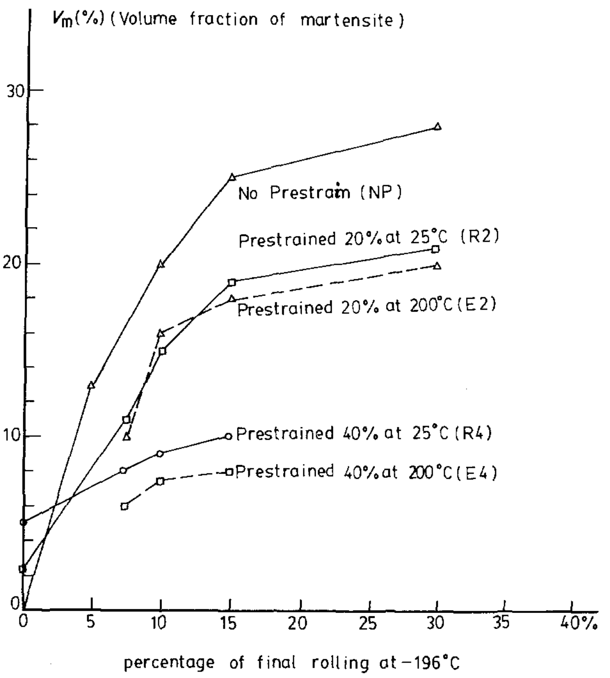

Figure 3 Correlation between the volume fraction of martensite and the amount of prestrain and final strain in specimen 1N.

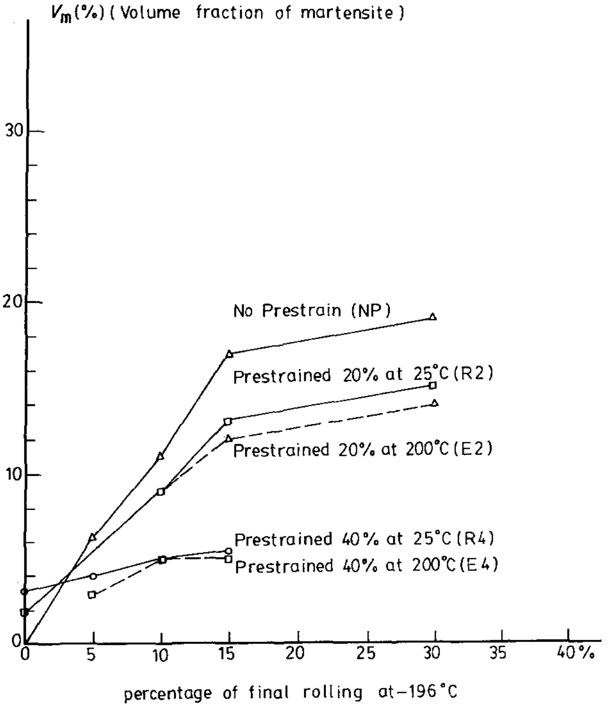

Figure 4 Correlation between the volume fraction of martensite and the amount of prestrain and final strain in specimen NI.

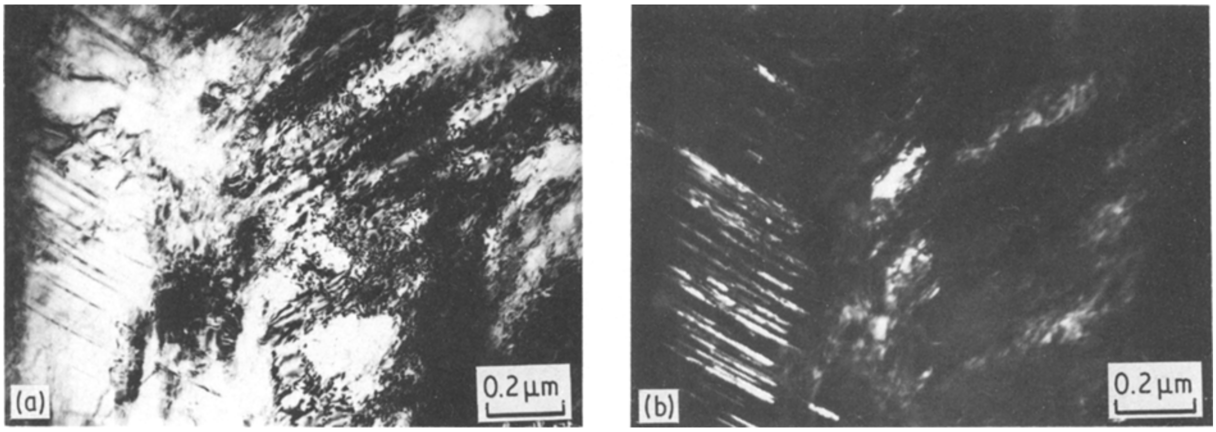

Figure 5 TEM microstructure of specimen NI-R2-15 which shows that the ε growth was hindered by high dislocation density. (a) Bright-field image, (b) dark-field image due to ε reflection.

The main difference between Figs 2, 3 and 4 is the amount of martensite volume fraction. Comparing the amount of martensite at the same prestrain and at the same final strain, we found that alloy NO always possessed the largest amount of martensite and alloy NI always possessed the least amount of martensite. This means that the order of austenite stability is NI > 1N > NO. The enhancing effect of alloying elements (nickel and nitrogen) on austenite stability is evident. Nickel demonstrated a more pronounced effect than nitrogen. If prestrained 20% at 200° C (E2) the martensite volume fraction is 23%, 20% and 14%, respectively, in alloys NO, 1N and NI at a final strain of 30%. If prestrained 40% at 200° C (E4) the martensite volume fraction is 11%, 7.5% and 5% in alloys NO, 1N and NI, respectively, at a final strain of 15%. The effect of prestrain on austenite stability was due to the fact that the growth of martensite was hindered by a variety of substructures. The hindering effect of martensite growth at grain boundaries and twin boundaries was demonstrated before [5]. Fig. 5 shows that the ε martensite growth was hindered by high dislocation density.

4. Discussion

It is well established that the addition of nickel and nitrogen in alloy steels will increase austenite stability because nickel and nitrogen are both austenite formers. In addition, nickel has a stronger effect than nitrogen on austenite stabilization. Our experimental results were in agreement with the theory mentioned above. The M_s of the ε-phase in alloy NO was above − 196° C but that of the ε-phase in alloys 1N and NI was below − 196° C. Owing to the addition of nickel and nitrogen, not only the M_s was lowered but also the volume fraction of strain-induced martensite was decreased significantly, as shown in Figs 2 to 4. Alloy NI possessed the best austenite stability, alloy 1N had better austenite stability than alloy NO. This is in agreement with Graham and Youngblood's report [8] which indicated that the amount of ε-phase would decrease as nickel content was increased. Thus, the addition of nickel will increase stacking fault energy and stabilize the austenite.

In this examination, α-phase was observed only when accompanied by ε-phase, regardless of alloy type. The amount of α-phase was always much smaller than ε-phase. α-phase was found predominantly at the intersection of two ε bands. All our experimental results indicated that ε formation was mostly by a regular overlapping process of stacking faults [9] and the sequence of martensite transformation was $\gamma \rightarrow \varepsilon \rightarrow \alpha$ [5, 7, 10].

The improvement of austenite stability by prestrain was evident in this study, regardless of alloy type. The effect of prestrain was to introduce a high density of stacking faults, dislocation tangles, subcells and ε bands which retarded martensite growth so the austenite stability was enhanced. The effect of prestrain on austenite stability was more pronounced if the prestrain was done at 200° C rather than at 25° C, regardless of alloy type. The mechanism was explained in terms of the favouring effect of stacking faults on ε-phase nucleation and the hindering effect of substructures on ε-path growth as described previously [5].

5. Conclusions

1. The addition of nickel (2.43%) and nitrogen (0.11%) afforded significant improvement to austenite stability. Both nickel and nitrogen are austenite formers which can decrease M_s and increase austenite stability. The volume fraciton of strain (30% at − 196° C)-induced martensite decreased from 32% to 28% and 19%, respectively, due to the addition of nitrogen and nickel. If prestrain (40% at 200° C) was done, the volume fraction of strain (30% at − 196° C)-induced martensite decreased from 23% to 20% and 14%, respectively, due to the addition of nitrogen and nickel.

2. All experimental results indicated that ε-phase formation was mostly by a regular overlapping process of stacking faults and the sequence of martensite transformation was $\gamma \rightarrow \varepsilon \rightarrow \alpha$ regardless of alloy type.

3. The effect of prestrain on austenite stability was evident and the effect was more pronounced if the prestrain was done at 200° C rather then at 25° C, regardless of alloy type. The mechanism can be explained in terms of the favouring effect of stacking faults on ε-phase nucleation and the hindering effect of substructures on ε-phase growth [5].

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